



IN THE MATTER OF  
US PATENT APPLICATION NO.  
09/700,240  
FILED 12 DECEMBER 2000  
IN THE NAME OF BURLING et al

RECEIVED  
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TC 1700

**STATUTORY DECLARATION**

I, Dr Paul Jackson, of 172 Church End, Cherry Hinton, Cambridge CB1 3LB, United Kingdom; hereby declare as follows:

1. I was awarded a degree in Chemistry and Molecular Physics by the University of Nottingham, United Kingdom in 1997 and was subsequently awarded a Dr of Philosophy (PhD.) entitled "Reactive Sintering of Stainless Steel" by the School of Mechanical, Materials, Manufacturing and Engineering Management, University of Nottingham, United Kingdom in 2002. I am also an Affiliate member of the Institute of Materials, Minerals and Mining.
2. I have been employed by TWI Ltd., a company related to The Welding Institute the assignee for US patent application no. 09/700,240 (hereinafter the Application), since 2000. Currently, I hold the position of Senior Project Leader, Advanced Materials and Processes, and my responsibilities include research in the field of high temperature materials.
3. I am familiar with the subject matter of the Application, and have been involved in the testing and optimisation of products according to the Application, and in the commercialisation of those products under the trade name Barrikade.
4. I have read and understand the objections raised in the

Office Action dated 28 April 2003 issued by the United States Patent and Trademark Office, and I have read and understood the disclosure in British patent no. 693,168, to George Carpenter.

5. The two-step coating method disclosed by Carpenter differs from the two-step method claimed in the Application as Carpenter's method requires melting (more accurately softening above the glass transition temperature) of his coating material, ie. silicate of soda (also known as sodium silicate), in order to coat vermiculite particles, the melting being conducted in a rotary kiln. In contrast, the two-step method claimed in the Application does not require melting of the coating material, but instead the coating process is conducted at much lower temperature, typically around a room temperature, and requires as a final step heating and/or curing of the ceramic binder at significantly lower temperatures than those experienced in a rotary kiln.

6. In order to demonstrate the differences between the two methods, on the behalf of TWI I commissioned Thermograde Process Technology Ltd., of Staffordshire, United Kingdom (hereinafter "Thermograde") to reproduce the Carpenter method, using amounts of vermiculite and binder, and vermiculite particle size distribution, according to the invention claimed in the Application. It was necessary to commission Thermograde to carry out the reproduction of the Carpenter method because TWI Ltd. does not own a rotary kiln.

7. In order to give the best possible chance for the Carpenter method to work, the proportions of vermiculite and sodium silicate binder were selected so as to maximize the quantity of binder available to the system, resulting in 65% by weight sodium silicate in the dry product. The method was carried out in two stages, as follows:

### Stage 1

300 g vermiculite (Dupre Micafil supplied by Microfine Minerals, at least 80 weight % retained between 2.8 and 8 mm) was dry mixed with 555 g dry sodium silicate powder, and added to a rotary kiln. The rotary kiln was operated with a 800°C hot zone (ie. above the glass transition temperature of sodium silicate) and a rotation speed of 3 revolutions per minute.

Thermograde reported that adhesion of vermiculite granules to the inner tube of the kiln occurred with 10 minutes, and became worse until the tube became blocked after approximately one hour. The coated material had to be manually extracted from the tube. A small deposit remained in the tube which was fused quite strongly to its surface, but it was thought that it should be possible to remove this residue by immersion in dilute acid. On inspection, the vermiculite granules obtained could be seen not to be coated homogeneously with the silicate binder. The resulting product is shown in Figure 1 attached hereto.

### Stage 2

250 g of the pre-coated vermiculite obtained from Stage 1 was mixed with 380 g dry sodium silicate powder, in order to produce a final, dry, product containing 65% by weight sodium silicate.

The mixture was poured into a porous stain steel mould, to which uniaxial pressure was applied while heating to 800°C for three hours. On inspection, the product could be seen not to be bonded throughout its thickness. The resulting product is shown in Figure 2 attached hereto.

8. For comparison purposes, a two-stage process as claimed in the Application was carried out, using a slightly lower amount of sodium silicate binder (to give a dry product containing 53 weight % binder), and the same grade vermiculite. Similar results would have been achieved at higher amounts of sodium

silicate binder, and in particular at 65 weight % sodium silicate binder as used in the reproduction of the Carpenter method. The process was conducted as follows:

#### Stage 1

150 g as above vermiculite (Dupre Micafil supplied by Microfine Minerals, as above) was mixed with 225 g Barrikade 10 Binder (supplied by Forafix Ltd.) and dried at 100°C for two hours. Barrikade 10 Binder is a commercial liquid sodium silicate binder, and is the binder used in the commercial process according to the Application. On inspection of the product the vermiculite was homogeneously coated with binder. The product obtained is shown in Figure 3.

#### Stage 2

240 g of pre-coated vermiculite (obtained from Stage 1) was mixed with 195 g Barrikade 10 Binder and dried under pressure overnight at 100°C. The product obtained was bonded throughout its thickness, and is shown in Figure 4.

9. As is evident from a comparison of Figures 2 and 4, the products obtained by the two different methods are significantly different, the product obtained by the method of the Application having an integral structure bonded throughout its thickness. Furthermore, the product obtained by the Carpenter method could be broken manually, simply by hitting the product with the palm of the hand. In contrast, the commercial Barrikade product, made according to the Application, typically has a compressive strength in the range 550 to 600 kPa.

10. Furthermore, from a practical standpoint, in view of the difficulties encountered by Thermograde in reproducing the Carpenter method, and in particular the blocking of the tube of the rotary kiln, I doubt that the Carpenter method would be

commercially viable, at least in relation to the vermiculite used in the Application.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 USC 1001 and that such willful false statements may jeopardize the validity of the subject application or any patent issued thereon.

Declared by



Dr Paul Jackson

Dated

3<sup>rd</sup> NOVEMBER 2003

FIGURES

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Carpenter Method

Figure 1

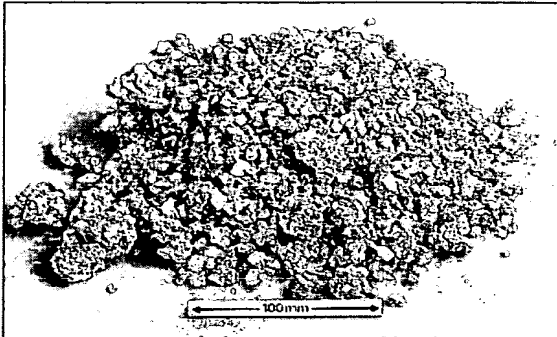
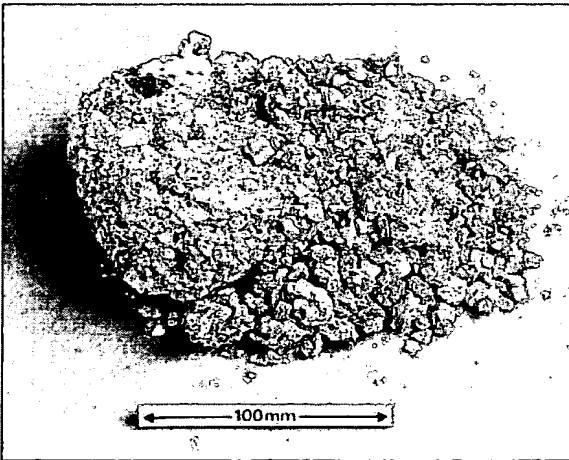
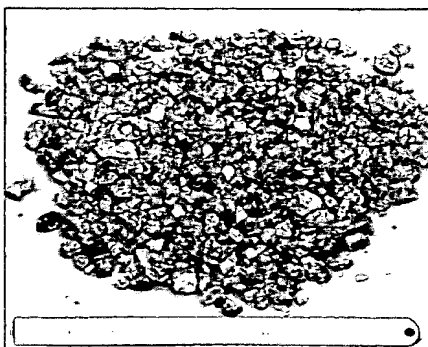


Figure 2



Method According to the Application

Figure 3

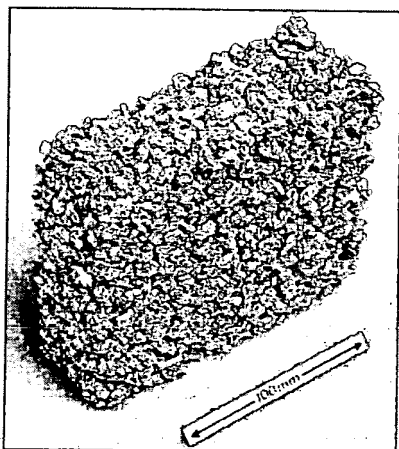


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Figure 4



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